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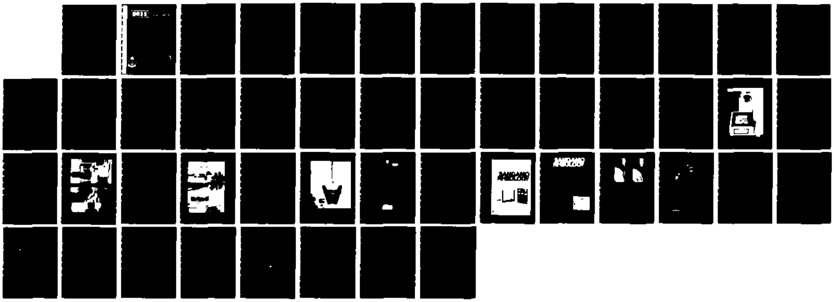

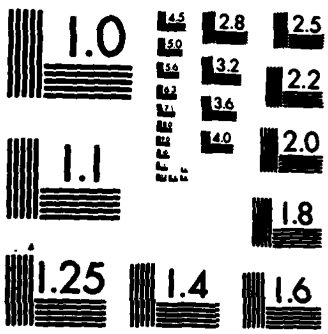

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NO. 377

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MEASUREMENTS OF ELONGATIONAL VISCOMETRY USING A FIBER SPINNING TECHNIQUE

PART I: MODIFICATIONS TO THE SANGAMO SCHLUMBERGER ELONGATIONAL VISCOMETER MODEL E4 (U)

by

M.D. Gauthier Mayer, W.J. Fenrick and S.J. Armour

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February 1984

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ABSTRACT

The Sangamo-Schlumberger Elongational Viscometer (Model E4), as delivered by the manufacturer, did not maintain a constant elongational load under constant flow conditions and consequently could not be used to accurately measure elongational viscosity. The extensive modifications made to the instrument at DRES to correct this problem are described in detail as are other improvements made to the instrument.

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ACKNOWLEDGEMENTS

The authors would like to thank
Professor A.B. Metzner
for many productive discussions
throughout the course of this work.

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INTRODUCTION

1. As part of its continuing study of methods of characterizing polymer solutions and of identifying the parameters important in the aerodynamic breakup of polymer solutions, the rheology group at the Defence Research Establishment Suffield (DRES) has undertaken a detailed study of the methods of measuring elongational viscosity. Three methods of measuring elongational viscosity (1) which were deemed to warrant detailed investigation were fiber spinning (2,3,4), Fano or syphon flow (5,6,7) and flow through porous media and/or convergent channels (8,9). Of these methods, that of fiber spinning was considered to show the most promise as it was the best documented (2,3,4) and as a commercial fiber spinning device, the Sangamo

Schlumberger Elongational Viscometer, was readily available. The other two methods would have required the design and construction of reasonably complicated instrumentation and the development of the associated software, both of which are relatively time consuming and labor intensive processes. Consequently, DRES purchased an Elongational Viscometer (Model E4) from Sangamo Schlumberger and had it commissioned by the Sangamo service representative, Mr. Roy Spooner on July 5, 1983.

2. As part of the purchase arrangement, the authors agreed to undertake a detailed evaluation of the viscometer and report to Sangamo any shortcomings discovered. The present report is a description of the tests undertaken, the problems encountered and the modifications made to the Sangamo Schlumberger Elongational Viscometer in order to render it capable of measuring the elongational viscosity of polymer solutions having zero shear viscosities in the range 1 to 100 poise. It is hoped that it will be of assistance to Sangamo in the manufacture of additional viscometers and the modifications of existent ones.

DESCRIPTION OF THE VISCOMETER

3. The Sangamo Schlumberger Elongational Viscometer is a fiber spinning device based on the design of Ferguson (4). The viscometer, which consists of a measuring chamber and a control unit, is shown in Figure 1 and described in the company literature included as Appendix I.

4. The operation of the viscometer can be briefly described as follows (see Figure 2). The viscometer is prepared for a run by first calibrating the transducer assembly for the range to be used during the run. The fluid whose viscosity is to be measured is placed in the fluid reservoir (No. 1) and the reservoir is pressurized slowly with compressed gas so that the delivery tube (No. 2) gradually fills with fluid. (Gradual filling is necessary to prevent the formation of bubbles in the line.) Once the fluid exits the nozzle (No. 3) uniformly, the nozzle is capped with a small circular piece of paper and the compressed gas is shut off. The piece of paper is held in place by atmospheric pressure, as the tube tries to drain back into the reservoir, and ensures that the delivery tube is completely filled with fluid. The elongational load is zeroed by adjusting the transducer coil position for gross adjustments and the zero control for fine adjustments. A weight corresponding to the desired full scale reading (1 g for the 100 range; 250 mg for the 25 range) is placed

on the top of the nozzle and the elongational load is set to read 1.00 using the full scale adjustment. This procedure is repeated until the expected readings are obtained at zero and at the full scale value of 1.00. Pressure is again applied and the resulting fluid flow causes the small piece of paper to fall off the nozzle as a fluid column (No. 4) forms at the nozzle exit. This column falls onto the drum (No. 15) which is rotating counter clockwise at a predetermined speed. The elongational load exerted on the fluid column as measured by the transducer assembly is recorded, the volumetric flow rate is determined and the fluid column is photographed for further analysis. Approximately ten minutes are required for each run.

5. In order to obtain accurate data, the following experimental parameters must remain constant once the run has started; a) the reservoir pressure which determines the volumetric flow rate, b) the elongational load as measured by the transducer assembly, c) the drum speed and drum position, and, d) the temperature of both the fluid and the viscometer itself. The viscometer must also be designed so that high resolution photography of the fluid column can be easily achieved.

APPARATUS TESTING

6. The authors started testing of the elongational viscometer on July 7 1983. The initial materials chosen were a silicone fluid, Viscasil 5000, supplied by Canadian General Electric; a silicone fluid, DC 200, supplied by Dow Corning; and Golden Shell 50 oil. Some relevant physical parameters of these fluids are given in Table I.

7. Preliminary tests with Viscasil 5000 indicated that the value of the elongational load did not remain constant once the run had commenced but continued to drift upward with time, never reaching a steady value. Table II gives elongational load – time data for a typical run using Golden Shell 50 oil. Figure 3 shows that a plot of elongational load as a function of time is linear with slope 2.23 mg/min and correlation coefficient $R^2 = 0.989$. At the end of the run, when the zero calibration was rechecked, with the delivery tube full of fluid and capped with a small piece of paper (as in the initial calibration procedure), the elongational load value was not zero as expected but was a positive number considerably larger than zero (see Table II).

8. Similar upward drifts in elongational load with time were observed for all three fluids tested. Table III gives the data obtained for 14 additional runs. In each case plots

of elongational load as a function of time were linear with the elongation load value not normally returning to zero at the end of the run. The numerical value of the slope, although not constant, changed over a relatively narrow range (0.88 – 2.79 mg/min). This observation coupled with the fact that the upward drift was observed for fluids of widely different viscosity and different density, implied that the drift was more likely an artifact of the measuring system than of the fluid used.

9. Possible causes of this increase in elongational load were:

a. variable volumetric flow rate during a run

b. variable drum speed

c. varying chamber and/or fluid temperature

d. bubbles in the fluid

e. incorrect alignment of the delivery tube – transducer assembly

f. electronic drift in the elongation load measuring assembly

Possibility (a) was ruled out as the volumetric flow rate, was determined at the start, middle and end of several runs (see Table III) and found to be constant during any particular run. Possibility (b) was also ruled out as the drift continued even when the fluid fell onto a stationary drum. Checks of the drum speed with a strobe light also indicated that it was constant. The temperature of the chamber was recorded during each run and found to vary less than 0.2°C during the course of a run. The fluid temperature in the reservoir was also monitored and found to be constant. The occurrence of bubbles in the fluid was also ruled out as a major factor as there was no evidence of bubbles in the fluid issuing from the nozzle once the run had commenced. It was also considered highly unlikely that a random occurrence such as bubble formation would lead to a systematic increase in elongational load with a variety of fluids and at several different operating pressures.

10. The possibility of incorrect alignment of the delivery tube-transducer assembly was examined in detail. The complete assembly was carefully reassembled and aligned using a cathetometer and the transducer was checked to make certain that the core was centered and free to move. The assembly was then carefully tightened down so that no slippage would occur during the run and the alignment rechecked. This operation had no effect on the elongation load, which still continued to drift upward with time.

11. The possibility of electronic drift in the elongational load measuring system was ruled out as the zero value of the elongational load did not drift appreciably with time. The possibility of a faulty amplification stage was also ruled out as the upward drift was observed on three different amplification ranges (100, 25 and 10 ranges).

12. The above considerations necessitated a detailed analysis of the mechanics of the measurement system (see Figure 2). When pressure was applied to the reservoir (No. 1), the fluid left the reservoir, passed along the delivery tube (No. 2) and formed a column (No. 4) at the exit of the nozzle (No. 3). The fluid column then fell onto the drum (No. 15). The added weight of the fluid column (No. 4) caused the nozzle to move downward. The downward movement of the nozzle resulted in downward movement, B_2, of the split column clamp (No. 5) which was transferred to the forward flexure mount hinge assembly (No. 6) by the Invar strip (No. 7) allowing downward movement, C_2, of the mount (No. 6). This downward movement, C_2, resulted in a greatly amplified upward movement of the transducer core (No. 8) in the direction D_2. The amplification factor between movement, C_2, and D_2 was determined to be approximately 10 to 1. The above analysis implied that if the apparatus was operating as expected, the weight of the fluid column would have been the only cause of the upward movement D_2 and the elongational load value should have remained constant once the fluid column had been established. Since the elongation load value did not remain constant but continued to drift upward with time, an additional factor, other than the weight of the fluid column, must have contributed to upward movement at D_2.

13. Further examination of the apparatus provided the answer. The main mounting columns (No. 9) had been machined down at the top to a diameter of approximately 0.4 inches so that they could be fitted into the upper cross member (No. 10) which was locked to the smaller portion of the column (No. 9) by the locking screws (No. 11). When the reservoir (No. 1) was filled with 500 grams of fluid and suspended from the cantilever mount (No. 12) the machined down portion of the mounting columns (No. 9) flexed backwards, as indicated by the arrow F. This backward flexing allowed the reservoir (No. 1) and cantilever mount (No. 12) to move downward as indicated by A. This downward motion at A resulted in an upward motion at B of twice the magnitude of that at A. Upward motion at B was directly transferred to C which in turn caused movement at D of 10 times the magnitude of that at C. If Figure 2 is examined, it is apparent that movement at A would result in a 20 fold movement at D. Therefore, if movement at A was 0.0001 inches the resulting movement at D would be 0.002 inches. Tests have shown that movement at D of only 0.001 inch will cause the elongational load to change by 30 mg (30 percent of the full scale value on the 10 range).

14. With the above analysis in mind the movements which occur during an actual run were considered. At the start of a run, the reservoir was filled with approximately 500 grams of fluid and suspended from the cantilever mount (No. 12) causing downward motion A. When pressure was applied to the reservoir, the fluid flowed along the delivery tube and out of the nozzle, forming the fluid column. Since the volumetric flow rate was constant throughout the experiment the reservoir was slowly emptied at a constant rate. As the reservoir was emptied, weight, in the form of fluid, was removed from it and the reservoir became lighter allowing the small section of the main column (No. 9) to recover from flexing by moving in the direction F_2. Slow steady upward movement at A then resulted in slow steady downward movement at B_2 and C_2 which in turn caused slow steady upward movement at D_2. Thus it appeared that the removal of fluid from the reservoir caused the steady upward drift in elongational load with time.

15. This hypothesis was verified by the following experiment in which the removal of fluid from the reservoir during a run was simulated by the removal of weights placed on the top of the reservoir. The reservoir and delivery tube were emptied of fluid and a total of 300 grams was placed on the top of the reservoir as shown in Figure 4. The elongational load was then zeroed. The weight was removed 10 grams at a time and the value of the elongational load recorded until all 300 grams had been removed. The data obtained is recorded in Table IV. Figure 5 shows a plot of elongational load as a function of weight removed. Although the data is not perfectly linear, there is a steady increase in elongational load as the weight is removed from the reservoir.

16. As a further verification, the data obtained in previous attempts to measure elongational viscosity was reduced to this form, by using the volumetric flow rate and fluid density to calculate the weight loss. Elongational load as a function of weight loss for runs 1, 10, 12, 13 and 15 of Table III was then plotted on Figure 5. The data from the weight removal experiment lies in the center of the narrow band of data obtained in the viscosity experiments.

17. Consequently, the hypothesis that the upward drift in elongational load was caused by the removal of fluid from the reservoir and the resultant straightening of the columns (No. 9) was verified.

DESIGN MODIFICATIONS

18. To correct this situation the reservoir was removed from the cantilever and mounted on the inner wall of the chamber surrounding the apparatus as shown in Figure 6 and Appendix II-1. A specially designed clamp (Appendix II-1, No. 6) was manufacturered to fit the upper portion of the reservoir (No. 5). This clamp was attached to an adaptor plate (No. 2) which was designed to fasten to the inner wall of the chamber (No. 1) using 4 existing screws (No. 3). A stainless steel flange (No. 7) was also manufactured which, when fitted to the top of the reservoir using the existing bolt hole pattern, not only sealed the reservoir but also allowed a 3/8 inch diameter stainless steel tube (No. 10) to pass through its center. A standard 3/8 inch Swagelok® fitting (No. 9) was drilled through and the center hole in the bolt hole pattern was enlarged to 7/16 inch to allow the tube to enter the reservoir. This stainless steel tube emerged from the top of the reservoir and was connected to the barbed hose insert (No. 13) by a length of 5/16 inch ID Tygon® pressure tubing (No. 12). Tygon® tubing was used because it is both flexible and transparent. Flexibility prevents the transfer of vibrations from the enclosure to the transducer assembly. Such vibrations could cause the transducer beam to resonate. Transparency permits monitoring of the fluid and the detection of any bubbles entrained in it. Since the viscometer would be used to test solutions known to undergo gelation if subject to high shearing stresses upstream of the nozzle, it was decided to eliminate any small diameter tubing in the fluid supply line between the reservoir (No. 4) and the delivery tube (No. 20). Consequently, the original 1/8 inch stainless steel supply tube was removed from the cantilever and a 1/4 inch N.P.T. tapped hole provided to accept the barbed hose insert (No. 13). In addition, the depth of the tap drill hole for the 3/8 inch B.S.P. thread in the front of the cantilever (No. 14) was extended until it reached the tap drill hole used for the 1/4 inch N.P.T. hole provided for the barbed hose insert. These modifications to the cantilever mount provided a supply line free of restrictions up to the B.S.P. fitting (No. 17). The B.S.P. fitting has a restriction incorporated into its design which reduces the duct to a diameter of 0.089 inches just prior to the entrance into the delivery tube (No. 20). The pressure transducer supplied with the apparatus is located in the electronics module of the system. For the experiments with the gelling solutions, the pressure is monitored just prior to the entrance to the delivery tube as this is the point closest to the nozzle where a transducer can be easily installed. To facilitate this measurement a 1/2 inch x 20 T.P.I. N.F. hole was provided in the top of the cantilever (No. 14) to accommodate a Bytrex Pressure Transducer (No. 15).

19. Since the completion of these modifications, the elongational load value has stopped drifting and remains essentially constant throughout the run. Table V gives elongation load – time data for a representative run using Golden Shell 50 oil. Figure 7 shows a plot of elongational load as a function of time for the data of Table V. Comparison of Figure 3, a typical run before modification, and Figure 7, a typical run after modification, graphically illustrates that the modification has permitted the achievement of a steady value for the elongational load.

20. Details of the components required for this modification are given in Appendix II.

21. Attention was now directed toward the design of a safety clamp for the transducer assembly and to methods of measuring the fluid column.

22. Since the alignment of the delivery tube-transducer assembly is quite time consuming and since the Invar strips and the hinge assembly are very easily damaged, a safety clamp was designed to facilitate the changing of the nozzle. This clamp which is shown in Figure 8 and illustrated in Appendix II-5 is described as follows. The mounting bracket (Appendix II-5 No. 1) was designed to attach to two existing screws originally intended to hold the retaining frame for the glass in the top of the chamber. The remainder of the clamp assembly rotates about the axis A-A to allow it to be retracted when not required. The length of the unit B is fixed and is arranged so that in the down position the jaws (No. 4) are located on either side of the nozzle. When the hand wheel (No. 2) is moved downward on the thread of the support (No. 3) the tapered portion of the wheel contacts the upper portion of the holding jaws (No. 4) forcing both outward. The forward portions of the clamping jaws are simultaneously forced inward approaching the nozzle from either side. This system allows the nozzle to be clamped firmly with little or no displacement. When the hand wheel is moved upwards two springs (No. 5) force the holding jaws away from the nozzle so that the clamp assembly does not touch the nozzle during retraction. Using this safety clamp the nozzle could be easily changed without damaging the Invar strip or hinge assembly and without changing the alignment of the delivery tube.

23. The fluid column (see Figure 2, No. 4 and Figure 9) can be analyzed either by measuring its diameter as a function of length during the run using a cathetometer or by photographing the column (4). Analysis during the run using the cathetometer was not

regarded as practical because of the long time required to take a sufficient number of data points to accurately define the fluid column. Any minor motions or fluctuations in the column would produce substantial errors in the measurements. This method also has the major disadvantage of not giving a permanent record of the column profile.

24. Photography has the dual advantage of giving a permanent record of the column profile at a particular instant in time and of requiring only a short period of time to take several exposures. Since the fluid column could most easily be viewed through the insulating door on the left hand side of the measuring chamber (see Figure 1) it was decided to photograph through this door. The enclosure was modified for photography in the following manner. A scale was positioned in the same plane as the fluid stream in order to provide a calibration for the photographs. The main door on the front of the chamber was covered with drafting paper to act as a diffuser for the flash. Strips of white cardboard were placed directly behind the fluid column at the side opposite the flash and on the inside of the main door to deflect the flash and illuminate the fluid column. Since the present experiments were conducted at room temperature it was possible to remove the left inside window leaving the outer, insulating door to close the chamber. This window is often splashed by fluid thrown off the drum and must be cleaned after each run, an operation which is difficult to do without disturbing the delivery tube assembly. The outer insulating door, however, opens allowing easy access for cleaning.

25. The equipment used consisted of a Nikon 35 mm SLR camera fitted with a 200 mm micro-Nikkor lens and equipped with a 200 W · s flash. A typical photograph obtained with this system is shown in Figure 9.

SUMMARY AND CONCLUSIONS

26. Preliminary evaluation of the Sangamo Schlumberger Elongational Viscometer E4 showed that the viscometer, as delivered by the manufacturer, could not be used to accurately measure the elongational viscosity of fluids having zero shear viscosities in the range 1 to 100 poise.

27. The principal problem with the viscometer was the fact that the value of the elongational load, which should remain constant once a measurement has started, continued to drift upward with time, never reaching a steady value.

28. The cause of this upward drift was the fluid delivery system; specifically, the method of coupling the fluid reservoir to the delivery tube-transducer assembly.

29. A new fluid delivery system was designed which eliminated this problem. Details of its construction are supplied so that other users of the Sangamo Schlumberger Elongational Viscometer can make the same modification.

30. The Sangamo Schlumberger Elongational Viscometer with the modifications made by the authors appears to be capable of accurately measuring the elongational viscosity of a variety of fluids.

31. The measurement of elongational viscosity of polymer solutions of interest to DND using this instrument is currently underway and will be the subject of part two of this report.

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TABLE I

RELEVANT PHYSICAL PROPERTIES OF THE NEWTONIAN FLUIDS TESTED

	DENSITY (g/mL) @ 25°C	VISCOSITY (poise) @ 25°C
Viscasil 5000 Silicone Fluid Lot BJ108	0.9702	72.6
Dow Corning 200 Lot AA6659	0.9657	1.96
Golden Shell 50 Oil 427-005-37	0.882	5.2

TABLE II

TYPICAL DATA FOR GOLDEN SHELL 50 OIL

BEFORE MODIFICATION OF THE VISCOMETER

100 range (1000 mg Full Scale)

TIME (min)	CHAMBER TEMPERATURE (°C)	DRUM SPEED (rpm)	PRESSURE (bar)	ELONGATIONAL LOAD (mg)
Zero calibration	24.0			0.00
0	24.0	701	0.52	35
2	24.0	702	0.57	39
3	24.0	702	0.56	43
4	24.0	702	0.56	46
5	24.0	702	0.57	47
6	24.0	702	0.57	49
7	24.1	702	0.57	51
8	24.1	702	0.57	53
9	24.1	702	0.57	55
10	24.1	702	0.57	58
zero calibration check	24.1			18

TABLE III

ELONGATIONAL LOAD VS TIME DATA FOR THE NEWTONIAN FLUIDS

RUN NO.	FLUID	LOAD RANGE	R^2	INTERCEPT (mg)	SLOPE (mg/min)	FINAL ZERO (mg)	VOLUME FLOW RATE (mL/sec)
1.	Viscasil	25	0.921	6.25	1.91	23.5	0.142
2.	5000	25	0.993	30.0	1.28	− 1.25	—
3.	Silicone	25	0.992	41.3	1.52	5.50	—
4.	Oil	25	0.997	36.3	0.88	5.75	—
5.		25	0.969	43.8	1.03	30.6	—
6.		100	0.989	23.2	1.56	6.00	—
7.		10	0.998	31.2	1.37	− 11.3	—
8.		10	0.995	11.7	1.69	—	—
9.	Dow	10	0.968	29.9	1.99	− 9.9	—
10.	Corning	25	0.996	27.6	2.51	− 22.0	0.338
11.	200 Fluid	100	0.923	27.8	2.79	—	—
12.	Golden	100	0.989	25.0	1.97	—	0.282
13.	Shell	100	0.989	35.6	2.23	18.0	0.307
14.	50 Oil	25	0.995	14.8	2.19	—	—
15.		10	0.998	28.4	1.80	24.6	0.315

TABLE IV

ELONGATIONAL LOAD AS A FUNCTION OF WEIGHT REMOVED FROM THE TOP OF THE FLUID RESERVOIR

WEIGHT REMOVED (g)	ELONGATIONAL LOAD (mg)	WEIGHT REMOVED (g)	ELONGATIONAL LOAD (mg)
0	0.00	140	19.9
1	1.0	150	20.9
3	1.4	160	22.1
5	1.8	170	23.1
10	2.0	180	24.2
20	3.2	190	25.4
30	4.4	200	26.5
40	6.2	210	27.4
50	7.2	220	28.4
60	8.3	230	29.1
70	9.3	240	30.4
80	10.8	250	31.1
90	12.2	260	32.6
100	13.0	270	33.3
110	15.6	280	34.4
120	17.4	290	36.4
130	18.7	300	37.3

TABLE V

TYPICAL RUN USING GOLDEN SHELL 50 OIL

AFTER MODIFICATION OF THE FLUID RESERVOIR POSITION

10 range (100 mg Full Scale)

TIME (min)	CHAMBER TEMPERATURE (°C)	DRUM SPEED (rpm)	PRESSURE (bar)	ELONGATIONAL LOAD (mg)
0	24.6	717	1.86	25.8
1.5	24.6	717	1.86	26.0
2	24.6	717	1.86	26.1
3	24.6	717	1.85	25.9
4	24.6	717	1.85	26.1
5	24.6	717	1.85	26.3
7	24.5	717	1.85	26.3
7.5	24.5	717	1.85	26.3
9	24.5	717	1.85	26.3
10	24.5	717	1.85	26.4

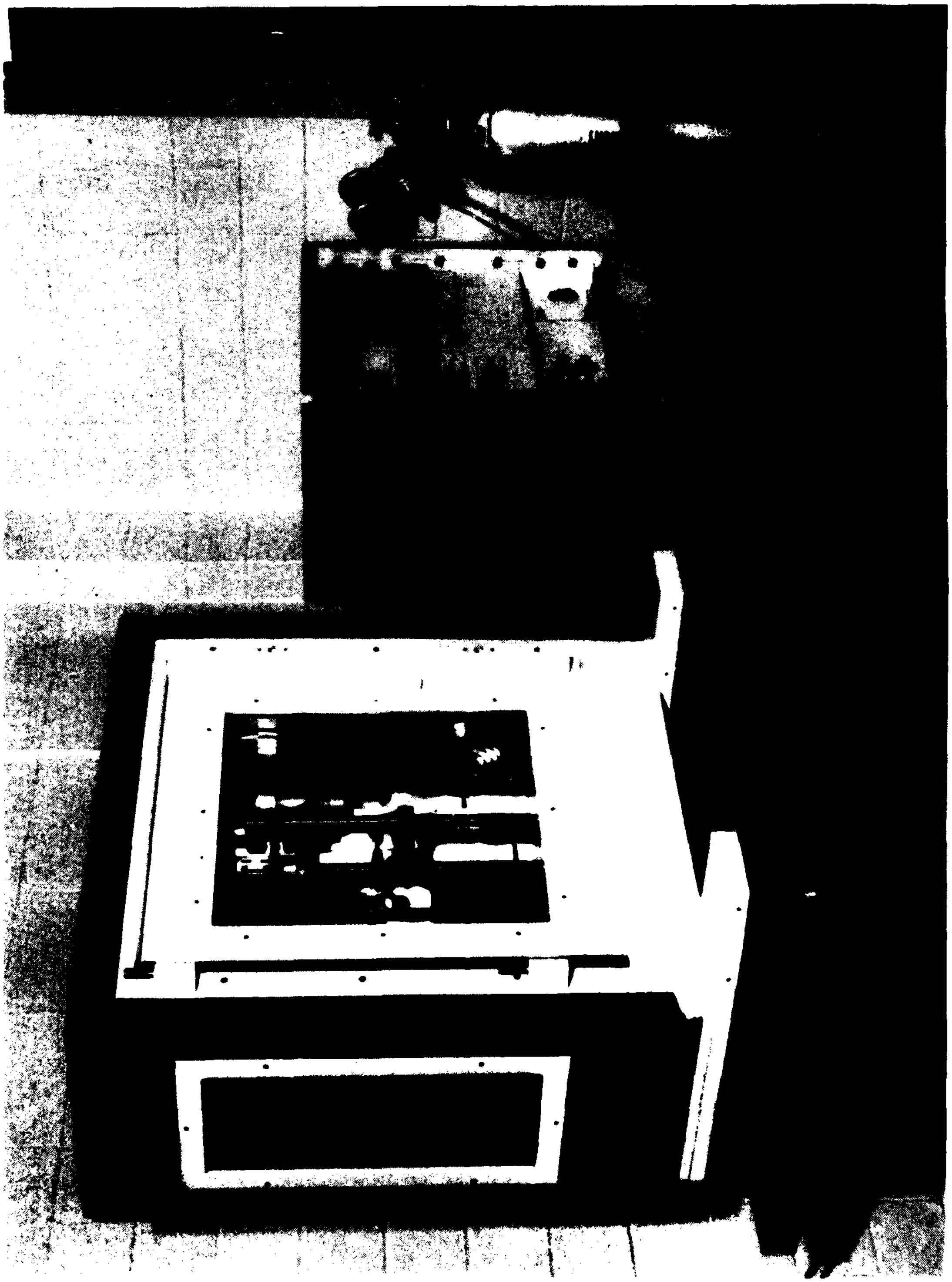
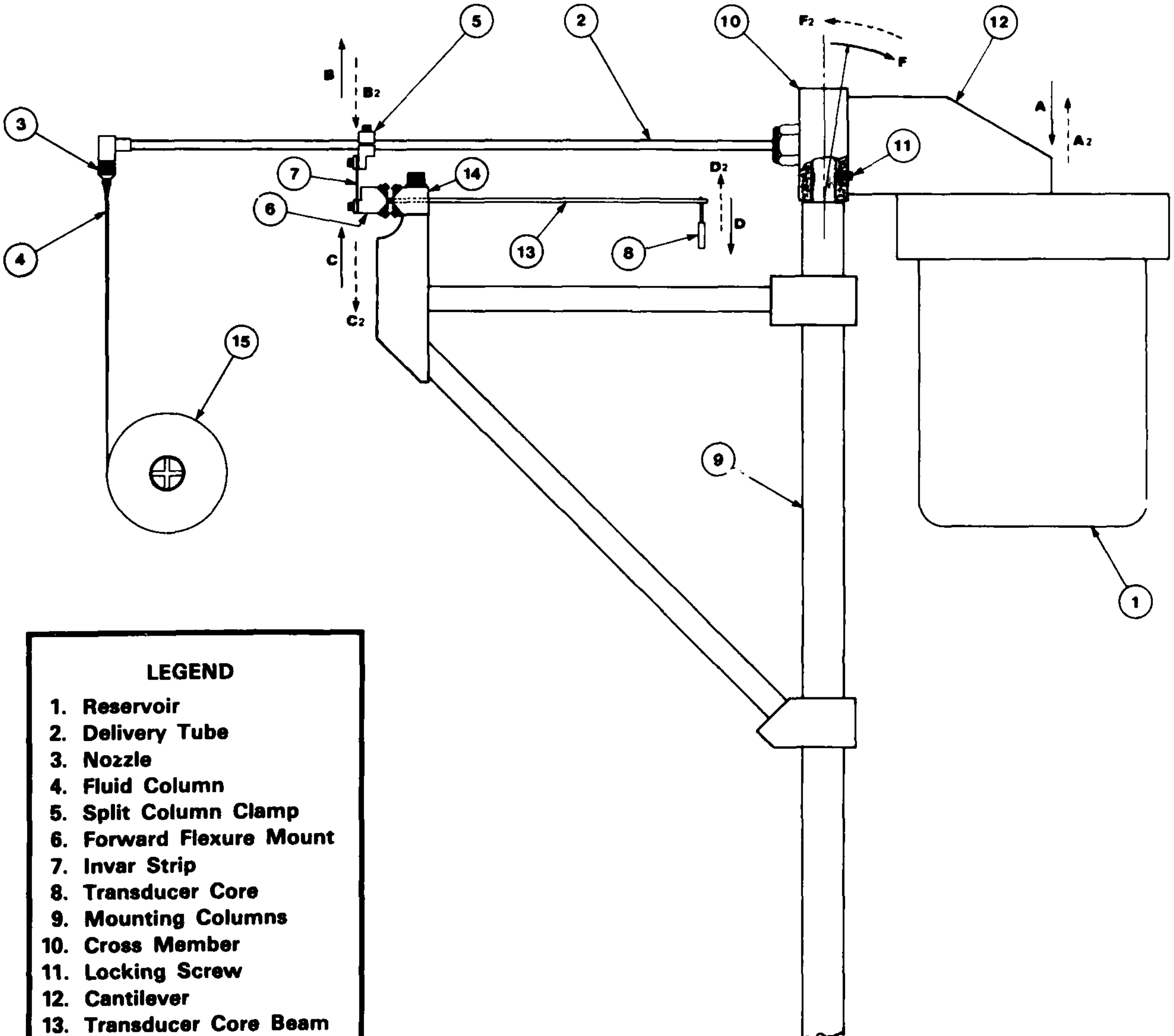


Figure 1

SANGAMO SCHLUMBERGER ELONGATIONAL VISCOMETER (Model E4)



LEGEND

1. Reservoir
2. Delivery Tube
3. Nozzle
4. Fluid Column
5. Split Column Clamp
6. Forward Flexure Mount
7. Invar Strip
8. Transducer Core
9. Mounting Columns
10. Cross Member
11. Locking Screw
12. Cantilever
13. Transducer Core Beam
14. Rear Flexure Mount
15. Drum

Figure 2

SCHEMATIC OF THE SANGAMO SCHLUMBERGER ELONGATIONAL VISCOMETER E4, SHOWING THE INTERIOR OF THE MEASURING CHAMBER

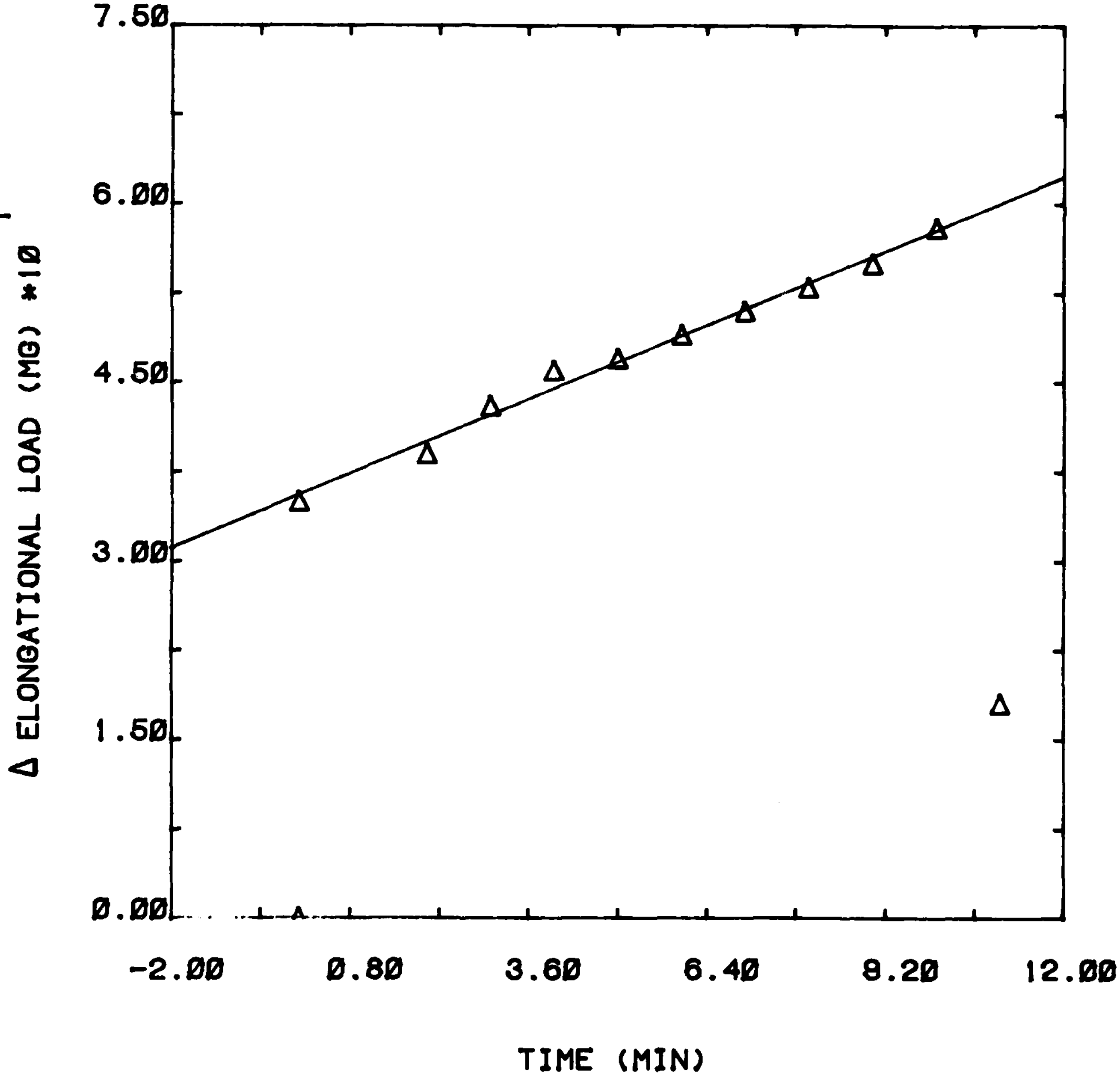


Figure 3

ELONGATIONAL LOAD VS TIME FOR GOLDEN SHELL 50 OIL BEFORE MODIFICATION OF THE VISCOMETER

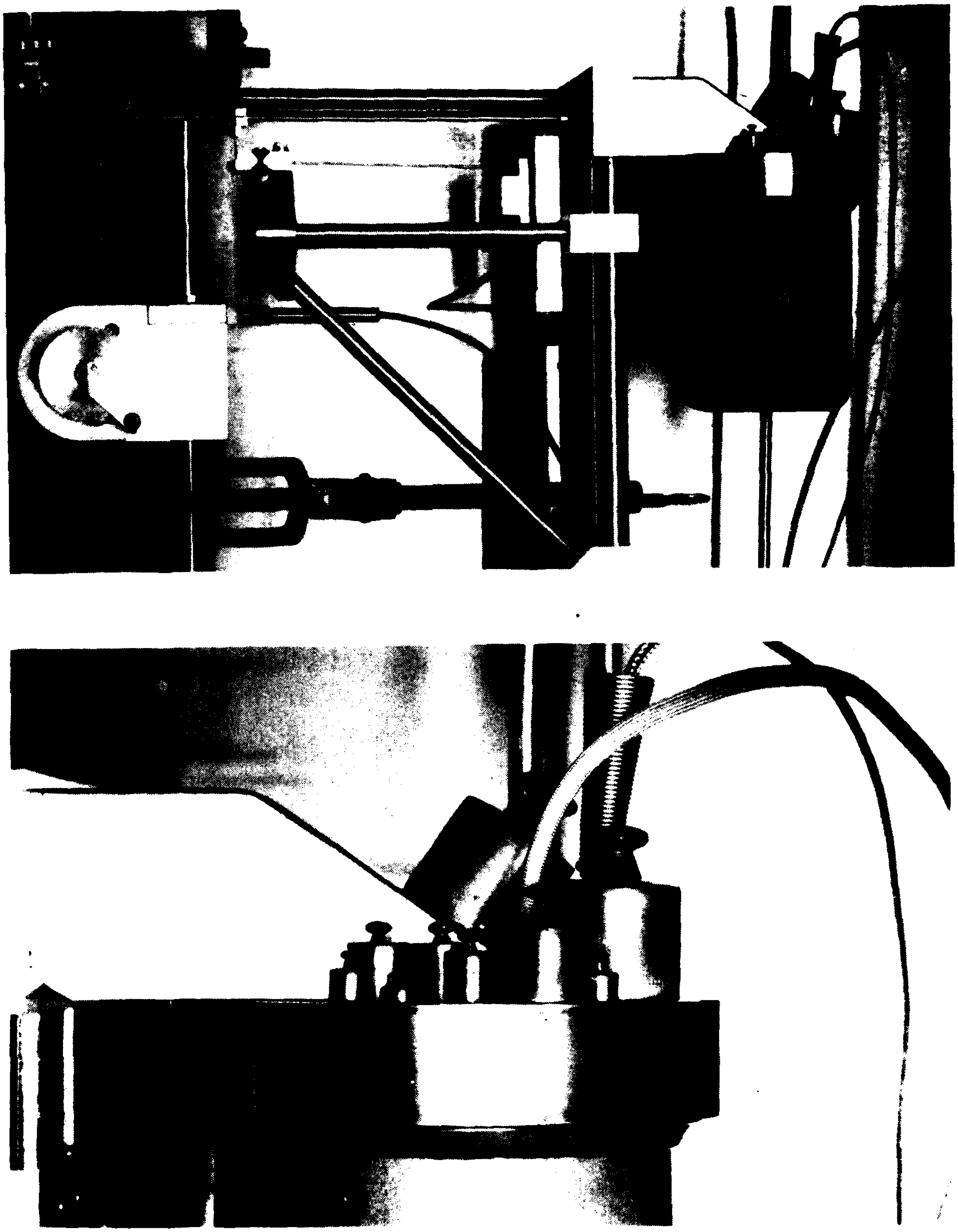
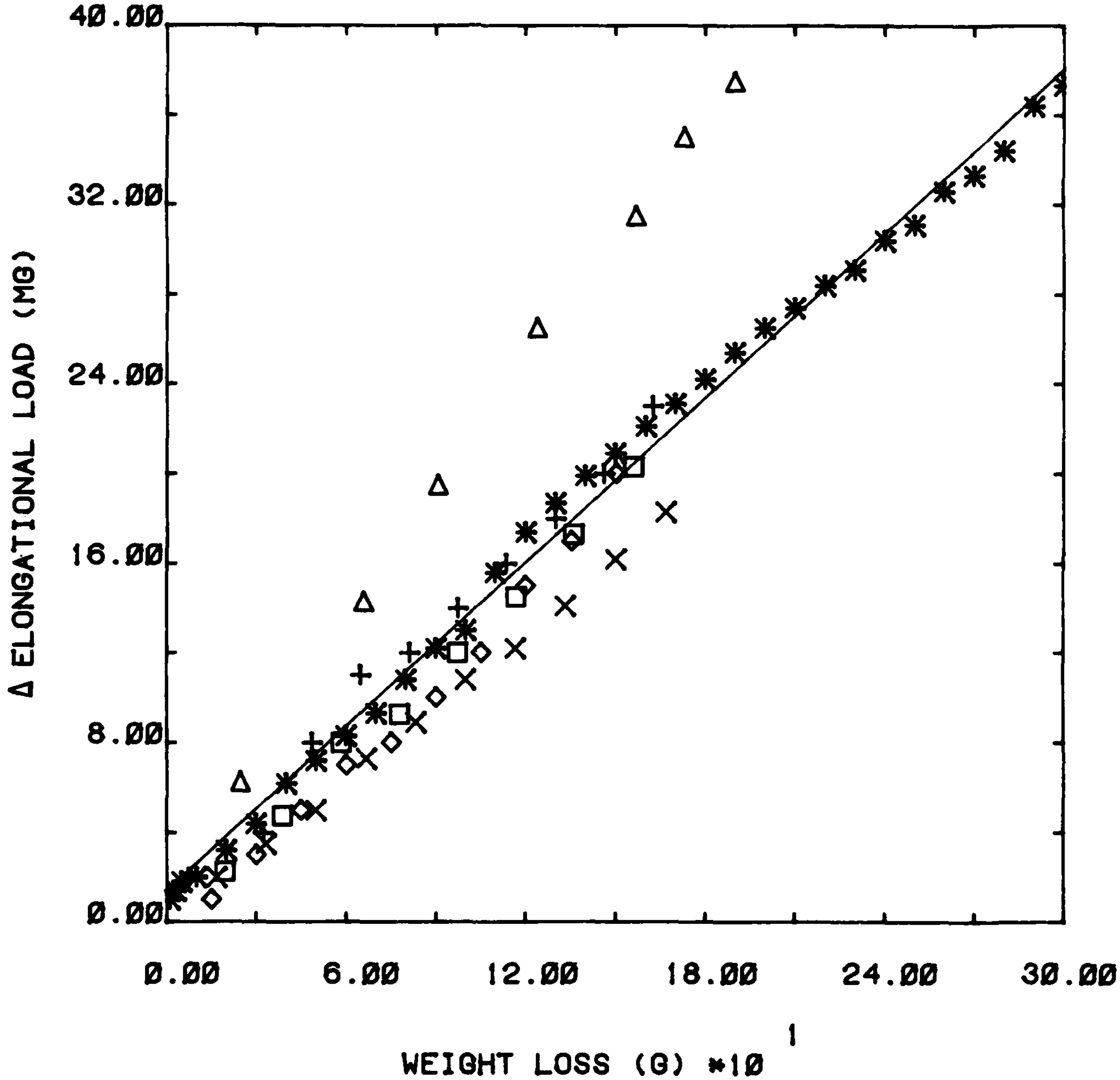


Figure 4

OVERALL AND DETAILED VIEW OF WEIGHTS PLACED ON TOP OF THE FLUID RESERVOIR



* Removal of Weights from top of Fluid Reservoir
△ Viscasil 5000 Silicon Fluid (Run #1)
□ Dow Corning 200 Fluid (Run #10)
◇ Golden Shell 50 Oil (Run #12)
\+ Golden Shell 50 Oil (Run #13)
× Golden Shell 50 Oil (Run #15)

Figure 5

ELONGATIONAL LOAD AS A FUNCTION OF WEIGHT LOSS

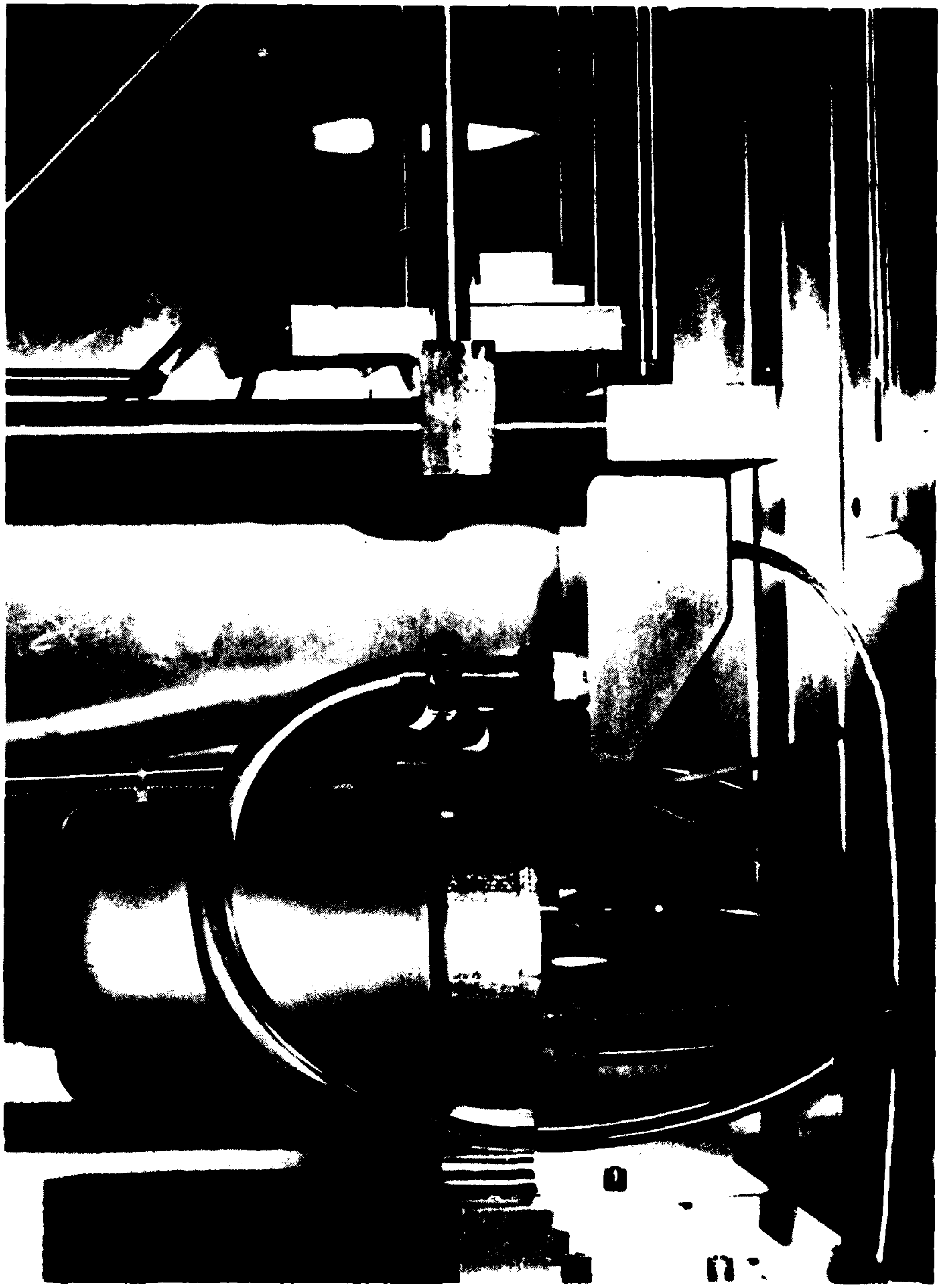


Figure 6

VIEW OF THE MODIFIED MEASURING CHAMBER, SHOWING THE SIDE MOUNTING OF THE FLUID RESERVOIR

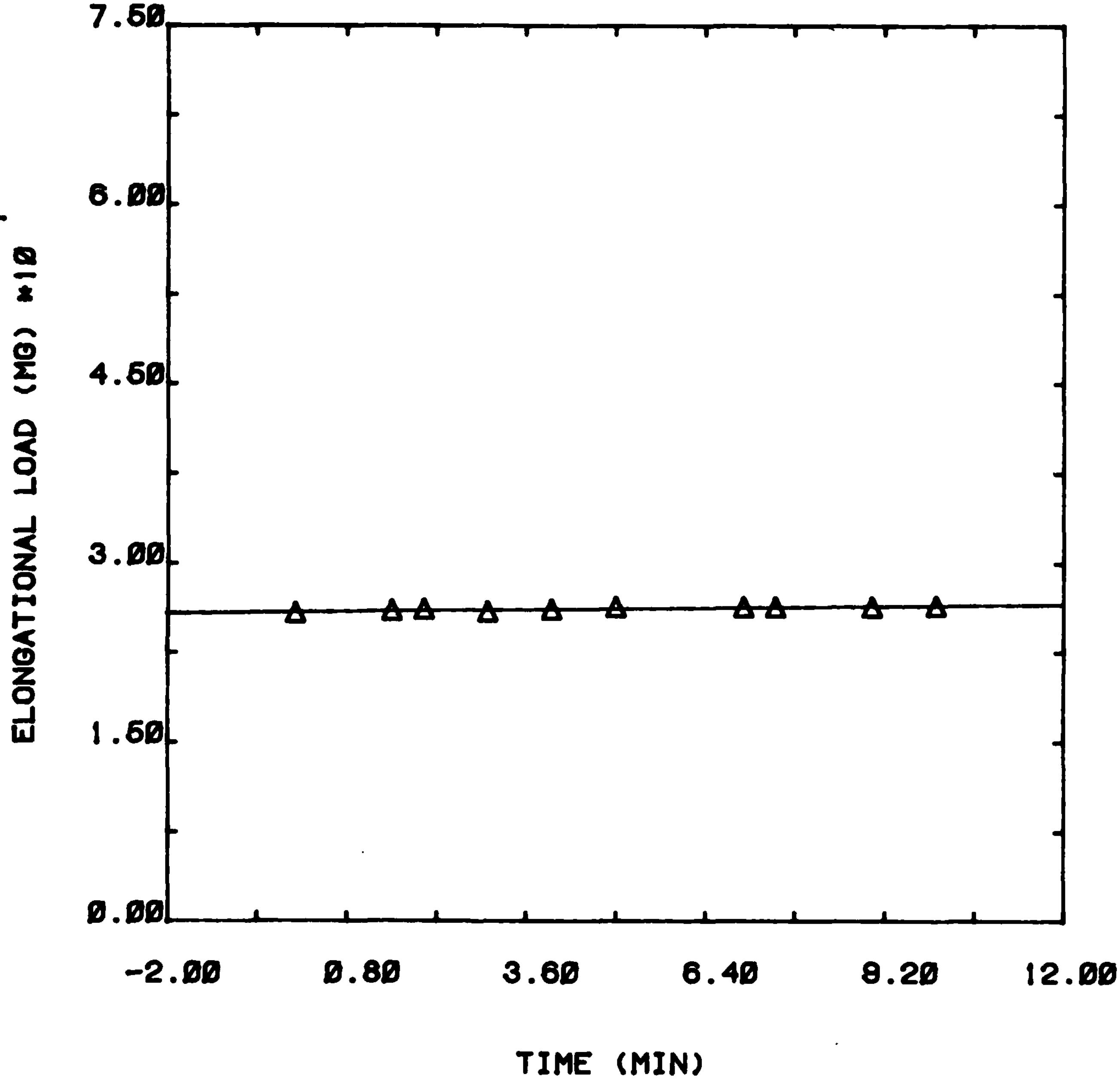


Figure 7

ELONGATIONAL LOAD VS TIME FOR GOLDEN SHELL 50 OIL AFTER MODIFICATION OF THE VISCOMETER

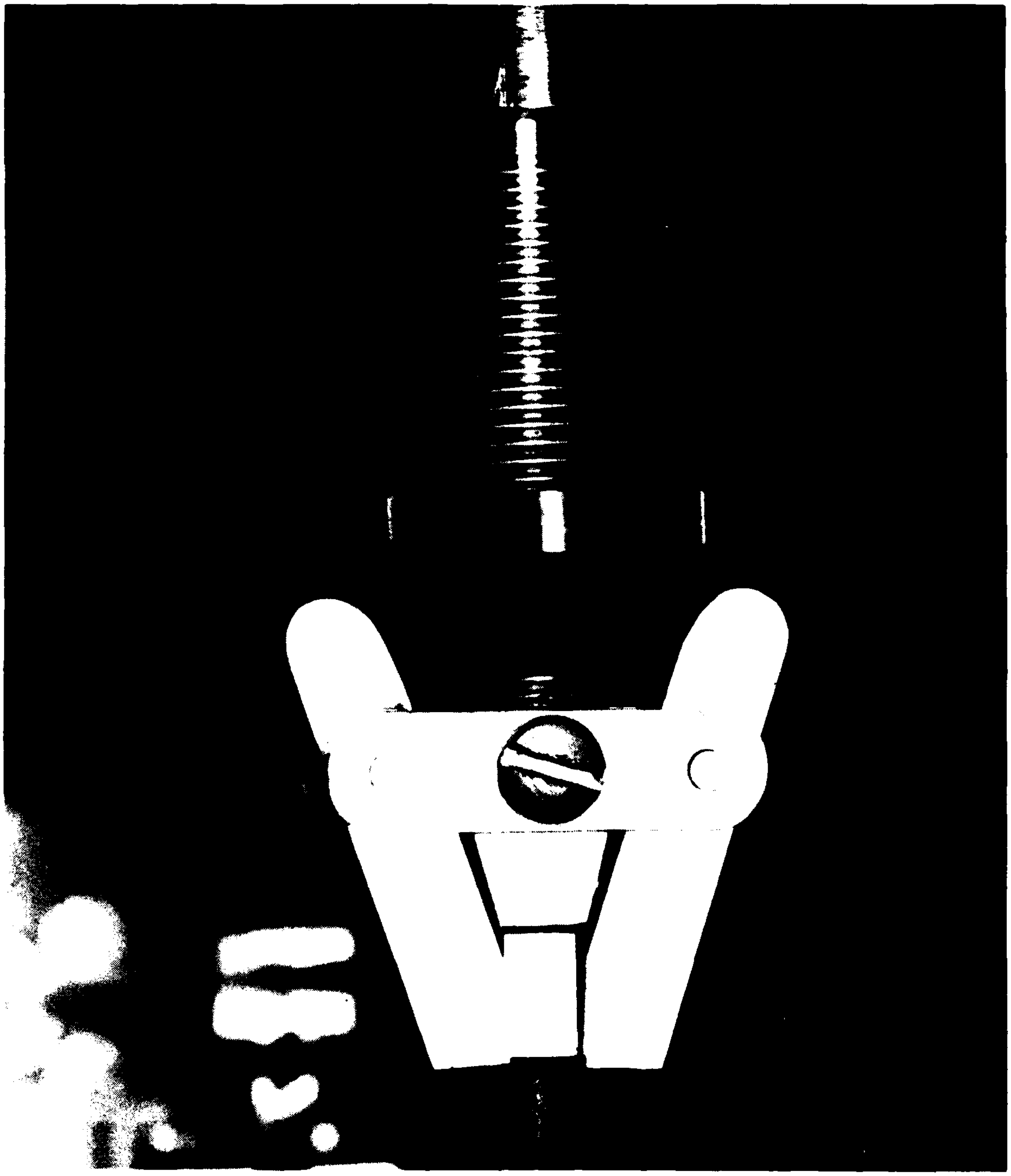


Figure 8

DETAIL OF THE SAFETY CLAMP ASSEMBLY FOR THE DELIVERY TUBE

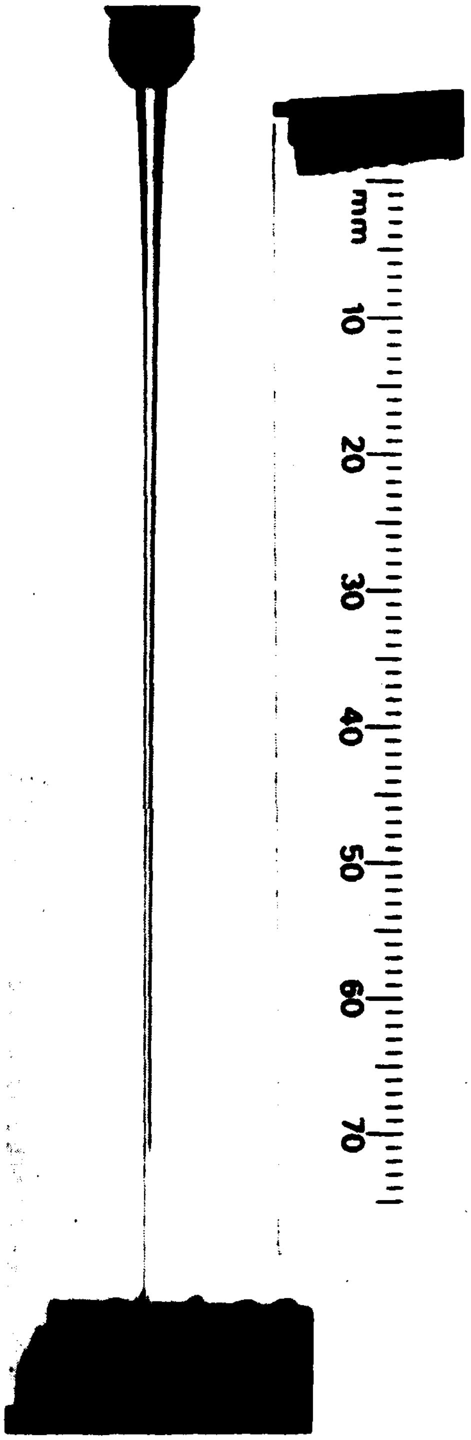
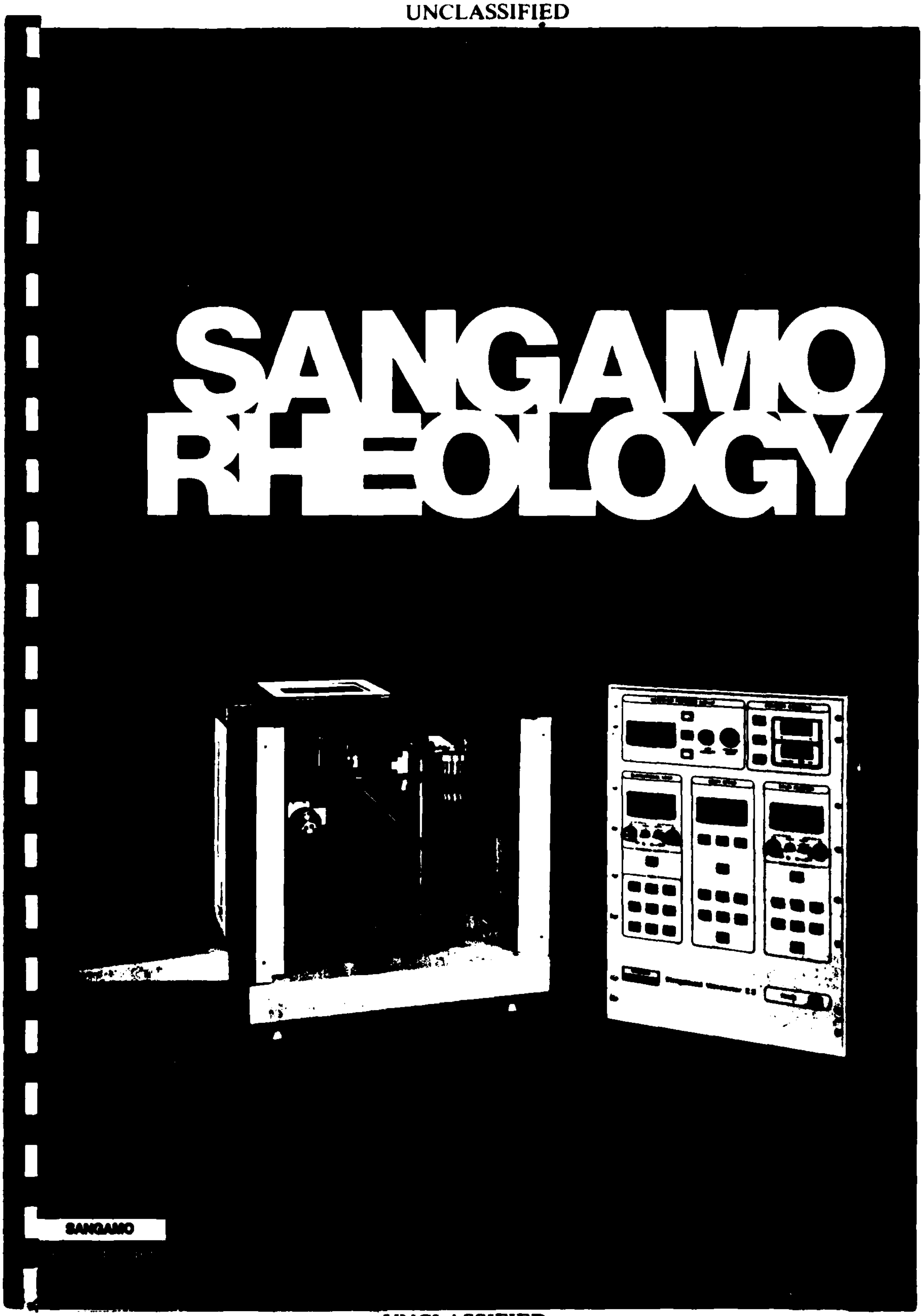


Figure 9

TYPICAL PHOTOGRAPH OF THE FLUID COLUMN, USING A 35 mm SLR CAMERA WITH A 200 mm MICRO-NIKKOR LENS AND A 200 W·s FLASH

APPENDIX I

SANGAMO SCHLUMBERGER BROCHURE
ON
ELONGATIONAL VISCOMETER


SANGAMO
RHEOLOGY
SANGAMO

SANGAMO RHEOLOGY

Rheology is the science of the deformation and flow of matter. The Rheology Division of Sangamo Schlumberger has been created to provide industry and research with the equipment necessary to advance the existing rapid growth in this science. The division is based on over 30 years experience with the Weissenberg Reogoniometer, an instrument which has contributed more than any other to further scientific development in this field.

The measurement of elongational viscosity under controlled laboratory conditions is increasing in significance as more industrial processes involve an elongational mode of deformation. In recognising this, Sangamo Schlumberger have once again pioneered the way by designing and developing an Elongational Viscometer capable of measuring the extremely small but vital loads created by the elongational deformation of low viscosity fluids. Technical development has been proceeding for a number of years based on a design by Dr J Ferguson of the University of Strathclyde, Glasgow, Scotland.

Reference: J Ferguson — Measurement of Elongational Viscosity of Polymer Solutions — Proceedings of the VIII International Congress of Rheology (1980).

APPLICATION

The technique of extruding a fluid and then extending it has generally been accepted for many years as the best known principle for establishing elongational viscosity. It avoids the difficulties of trying to stretch uniformly a comparatively low viscosity fluid. The information given by this method is rather different from that obtained using the constant rate of stretch (or constant load) equipment favoured by scientists using high viscosity fluids such as molten polymers. The term 'instantaneous elongational viscosity' has been used in certain cases. However, although theoretical development in this area is still in a state of rapid development, the implications for the technique in both industrial and academic research are clearly very great. Stretching flows represent a purer form of deformation than shear. The parameters controlling elongational viscosity and its variation with strain and rate of strain are currently not fully defined. The introduction of the Sangamo Schlumberger Elongational Viscometer therefore is most timely and should be seen as a powerful tool to further the science in this field.

Due to the lack of practical and theoretical data in this field it is impossible to define accurately a fluid range suitable for use with the instrument. For practical purposes it is essential that the sample fluid is 'spinnable' ie capable of forming a stable liquid filament in elongation. Further sections of this brochure have been set aside for a careful description of the operational functions and engineering ranges of the instrument. These ranges may be extended with practical experience and consultations with the Rheology Division of Sangamo Schlumberger are invited.

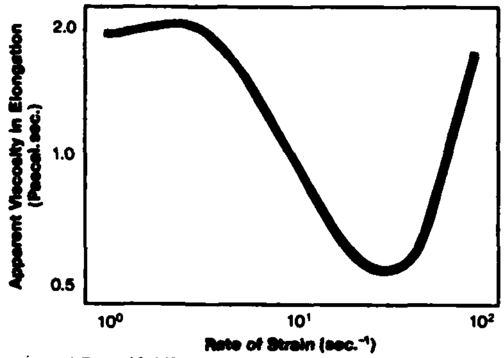


Figure 1 Test of 6.44% solution of Polybutadiene in Dekalin, showing a variation of apparent elongational viscosity with rate of elongation.

The instrument is known to be ideal for the measurement of polymer solutions and similar fluids in the following industries: *Oil, Food, Rubber, Paint, Pharmaceutical, Detergent, Polymer, Adhesive and Fibres.*

OPERATION AND MEASUREMENT PRINCIPLES

The Sangamo Schlumberger Elongational Viscometer consists of two main units — the measuring unit and the control and electronic readout unit.

Measuring Unit

The principle used is that of extruding fluid from a spinneret nozzle and extending it on a rotating drum. A diagrammatic interpretation is shown in Fig. 5.

The fluid is located in a stainless steel reservoir, which is pressurised by a clean gas such as air, nitrogen or carbon dioxide. The pressure, which is variable up to 3.5 Kgf/cm^2 forces fluid along a calibrated thin walled stainless steel tube and out through a spinneret nozzle. The fluid, falling vertically is picked up tangentially on a 50 mm diameter rotating drum which will cause it to elongate. The fluid is then cut away from the drum into a container below. Figs 3 and 4 clearly show the effect of the rotating drum in elongating the fluid. The length of the fluid filament is variable from 25 mm to 250 mm. This is achieved by mounting the drum on a precision slide and varying its position relative to the spinneret nozzle. An LVDT transducer for position sensing, and DC motor for slide movement are combined in a closed-loop system to provide the operator with an extremely accurate measurement and adjustment of filament length.

Figure 2 Measurement of Elongational Viscometer

Figure 3 Fluid under free fall from spinneret with drum static

Figure 4 Fluid in elongation with drum rotating

The force generated is measured by the deflection of the thin walled tube through which the fluid passes. The deflection is transferred through an invar strip, mechanically amplified via a crossed strip hinge, and measured by a frictionless LVDT transducer. The entire assembly has been designed for stability with mechanical stops to avoid overload damage. A range of tube wall thicknesses are available to obtain maximum sensitivity for any given fluid. The associated transducer amplifier offers a 400:1 range expansion and elongational load measurement down to 1 milligram is easily obtainable. Mechanical dmping is carried out by means of a paddle retained within silicon oil. An electronic filter is incorporated with nine instantly selectable cut off frequencies ranging from 2.5 Hz to 100 Hz.

Calibration is very simple, weights being placed immediately above the spinneret nozzle and electronic adjustments made accordingly. The simplicity of this operation allows calibration at any temperature level, thus avoiding concern over instrument coefficients.

The whole assembly is mounted on anti-vibration supports and housed within an environmental chamber. Level adjustment is provided to ensure vertical alignment of the filament to the drum. A powerful electric heating element is housed within the chamber and this is coupled to a three term controller. Circulation is fan assisted. The system is capable of providing a controlled temperature environment up to 100°C. The sensing element is mounted in the reservoir.

It is necessary to measure the diameter of the filament between the spinneret and the point of take-up on the drum. This is normally done using a vertically travelling microscope or cathetometer so that the diameter of the filament at any distance along the flow line can be determined. Alternatively this can be done photographically. The environmental chamber has glass panels to facilitate such measurements and mounting brackets are provided.

Control and Electronic Readout Unit

This is housed in a single console for bench mounting and easy operation. The panel is sub-divided into sections concerning various operating modes of the measuring unit. Direct readings are provided in digital form with associated voltage outputs available for external recording.

Reservoir Pressure: An indication of sufficient line pressure is provided together with a direct reading of reservoir pressure in Kgf/cm^2 with associated adjustment.

Chamber Controls: .. three term controller with pre-set temperature control and direct reading of reservoir (fluid) temperature.

Elongational Load: Direct reading of elongational load is provided up to 1 gramme with range expansion for greater sensitivity. Nine push button switches provide a range of filtering from 2.5 Hz to 100 Hz to eliminate high frequency vibrations. A section for the associated transducer calibration is included.

Drum Speed: Direct reading is provided for drum speed both in revs/minute or metres/second as selected. Push button controls are provided for fast and slow increase or decrease in the speed of rotation.

Drum Position: Direct reading of the filament length in millimetres is provided by measuring the drum position relative to the spinneret nozzle. Push button controls are provided for fast and slow increase or decrease of the filament length. A section for the associated transducer calibration is included.

ANCILLARY EQUIPMENT

A range of thin walled tubes is available to maximise sensitivity with any given fluid. These are supplied threaded for easy connection to the reservoir and also for interchangeable spinneret nozzles.

A range of spinneret nozzles is available with orifices from 0.3 mm to 2.0 mm diameter.

A single channel potentiometric chart recorder is available for direct connection to the elongational load output signal. An alternative multi-channel recorder can be used for continuously monitoring of other parameters.

Binary Coded Decimal (BCD) is available for all relevant functions thus providing outputs in digital form. Sangamo Schlumberger will be pleased to provide technical advice on interfacing with computer equipment.

A camera or travelling microscope is available for the measurement of the filament deformation.

SPECIFICATION

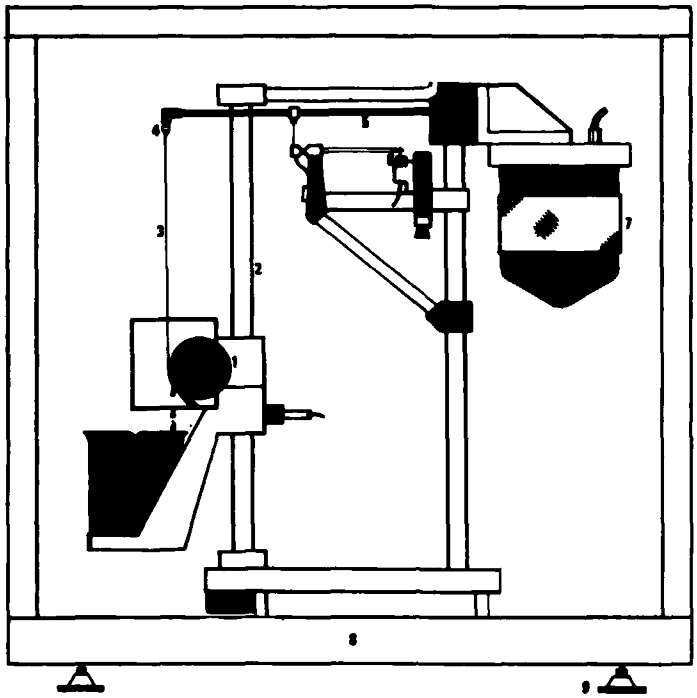


Figure 5 Diagram of Sangamo Schlumberger Elongational Viscometer Measuring Unit showing key components

1 Drum
2 Slide
3 Fluid filament
4 Spinneret
5 Thin walled tube
6 LVDT sensor
7 Reservoir
8 Environmental Chamber
9 Anti-vibration feet

Dimensions and weights
Measuring Unit
1000 mm wide 400 mm deep 725 mm high
Weight: 70 Kilogrammes

Control and Electronic Readout Unit (not illustrated)
500 mm wide 475 mm deep 725 mm high
Weight: 50 Kilogrammes

Mechanical

Reservoir
Stainless steel construction — capacity 500 ml

Fluid Pressure
Infinitely variable up to 3.5 Kgf/cm²
System exhausts to atmosphere at 4.22 Kgf/cm²
Reservoir overload pressure capability of 20Kgf/cm²

Elongational Load
Measuring range from 1 mg to 1000 mg
A range of calibrated stainless steel tubes are available with varying wall thicknesses — these provide maximum sensitivity for fluid under measurement
Damping, mechanically by paddle in silicon fluid and electrically with nine selectable ranges from 2.5 Hz to 100 Hz

Spinneret Nozzles
Stainless steel construction
Range of orifice diameters: 0.3, 0.5, 0.7, 1.0, 1.5, 2.0 mm

Filament
Length is infinitely variable from 25 to 250 mm
Diameter is measured by travelling microscope or photographically

Drum
Rotated by a closed loop drive system infinitely variable from 0–2000 revs/minute — equivalent to a surface speed from 0 to 5.24 metres/second
50 mm diameter stainless steel as standard. Other surface materials such as granite, ceramic, glass etc are available
A cutter is incorporated to remove fluid
Stroboscope speed measuring facility

Temperature
Environmental chamber with electric elements and fan circulation
Three term temperature control from ambient to 100°C monitored by platinum resistance thermometer in reservoir

Fluids
These must be 'spinnable' ie capable of forming a stable liquid filament in elongation

Electrical

Mains Input
115 V or 230 V ac +10% −15%, 48 to 65 Hz

Consumption
1500 W maximum

Outputs
The following analogue voltage outputs can be made available for external recording purposes. These are normally to a level of 1 V DC equivalent to the engineering units listed below (0.1 Ω typical impedance). Outputs for *Elongational Load* and *Filament Length* are available as standard. Binary Coded Decimal (BCD) outputs are available as options.

Fluid Pressure	1 Kgf/cm²
Elongational Load	1 g, 0.25 g, 0.1 g on selected range
Filament Length	250 mm, 100 mm on selected range
Drum Speed	1000 rpm (rotation) 1 metre/second (surface speed)
Temperature	100°C

Due to continuous improvements and changes in design we reserve the right to amend any specification without notice

SANGAMO
Schlumberger

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Designed by Atelier Marketing Services / Printed in England

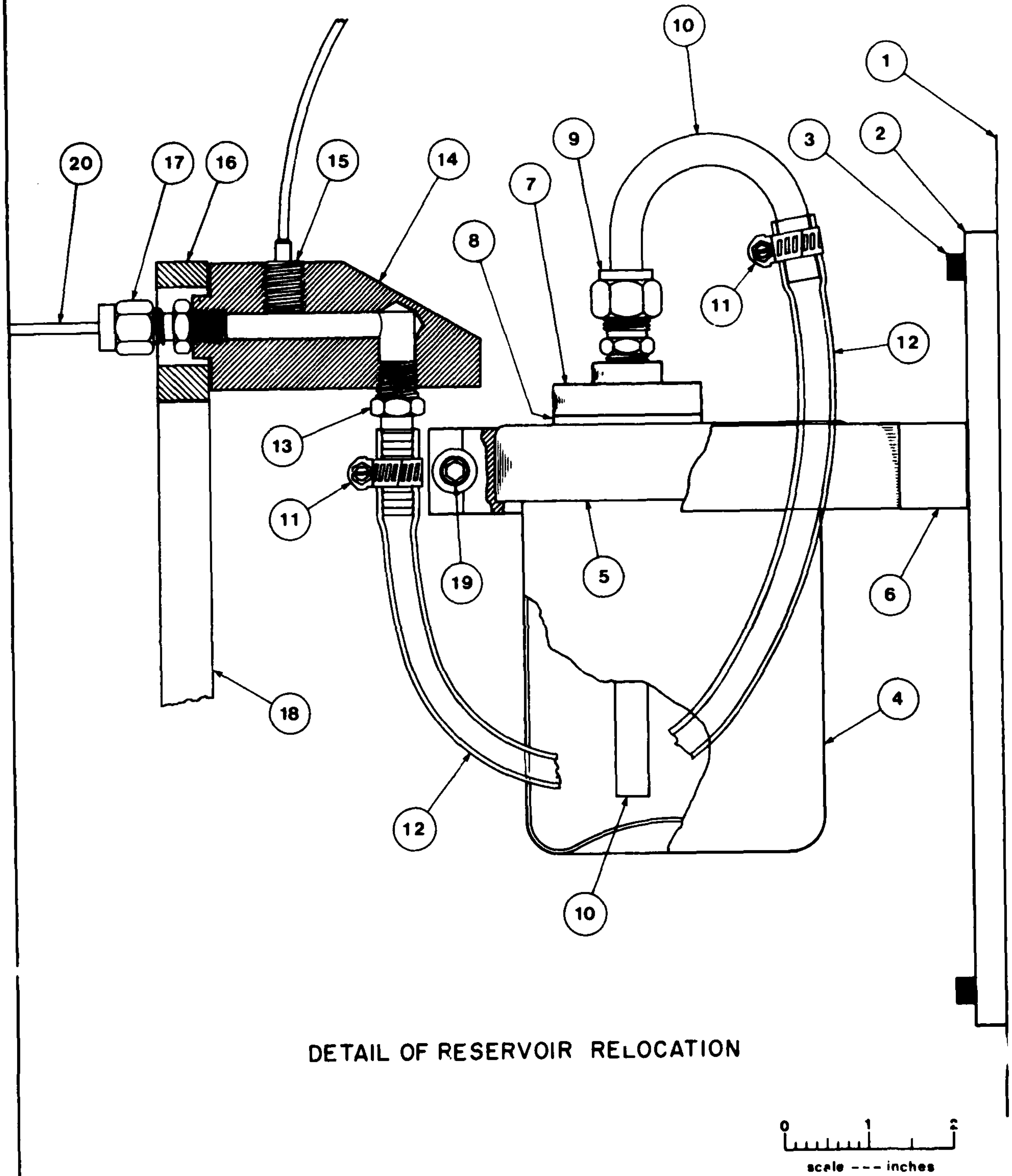
APPENDIX II

DRAWINGS OF DESIGN MODIFICATIONS

LEGEND, APPENDIX II-1

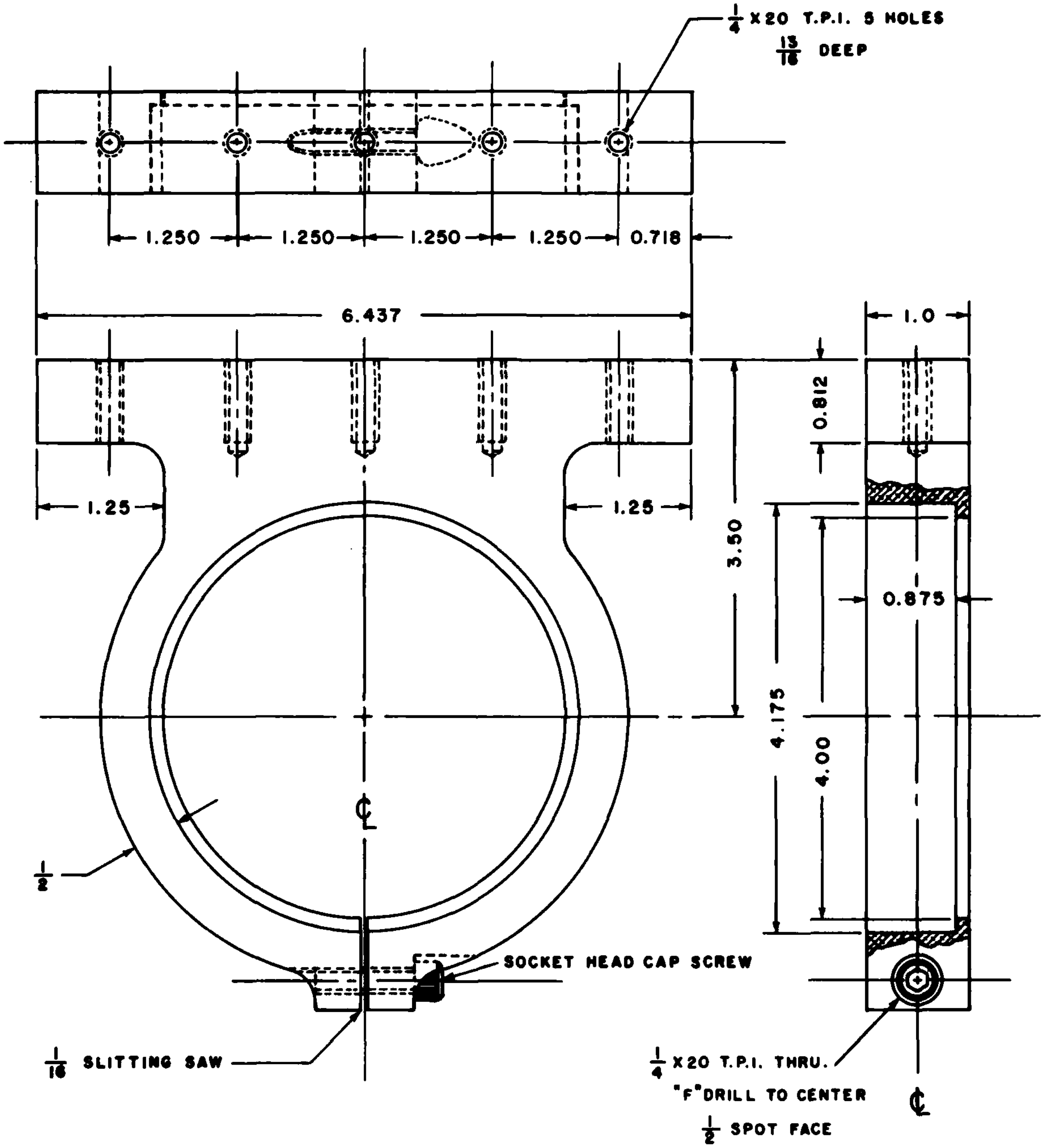
1. Inner Wall of Apparatus
2. Adaptor Plate
3. 8 × 36 UNF Socket Head Cap Screw
4. Reservoir
5. Reservoir Top
6. Reservoir Clamp
7. Stainless Steel Flange
8. Teflon® Gasket
9. Swagelok® Fitting (Drilled Through)
10. 3/8″ O.D. Stainless Steel Tubing
11. Tridon Hose Clamps
12. 5/16″ I.D. Tygon® Pressure Tubing
13. Barbed Hose Insert
14. Cantilever Mount
15. Bytrex Pressure Transducer
16. Cross Member
17. (Fitting) 3/8″ B.S.P. to 1/8″ O.D. Tubing
18. Mounting Column
19. 1/4 × 20 Socket Head Cap Screw
20. Delivery Tube

Appendix II-1



DETAIL OF RESERVOIR RELOCATION

Appendix II-2



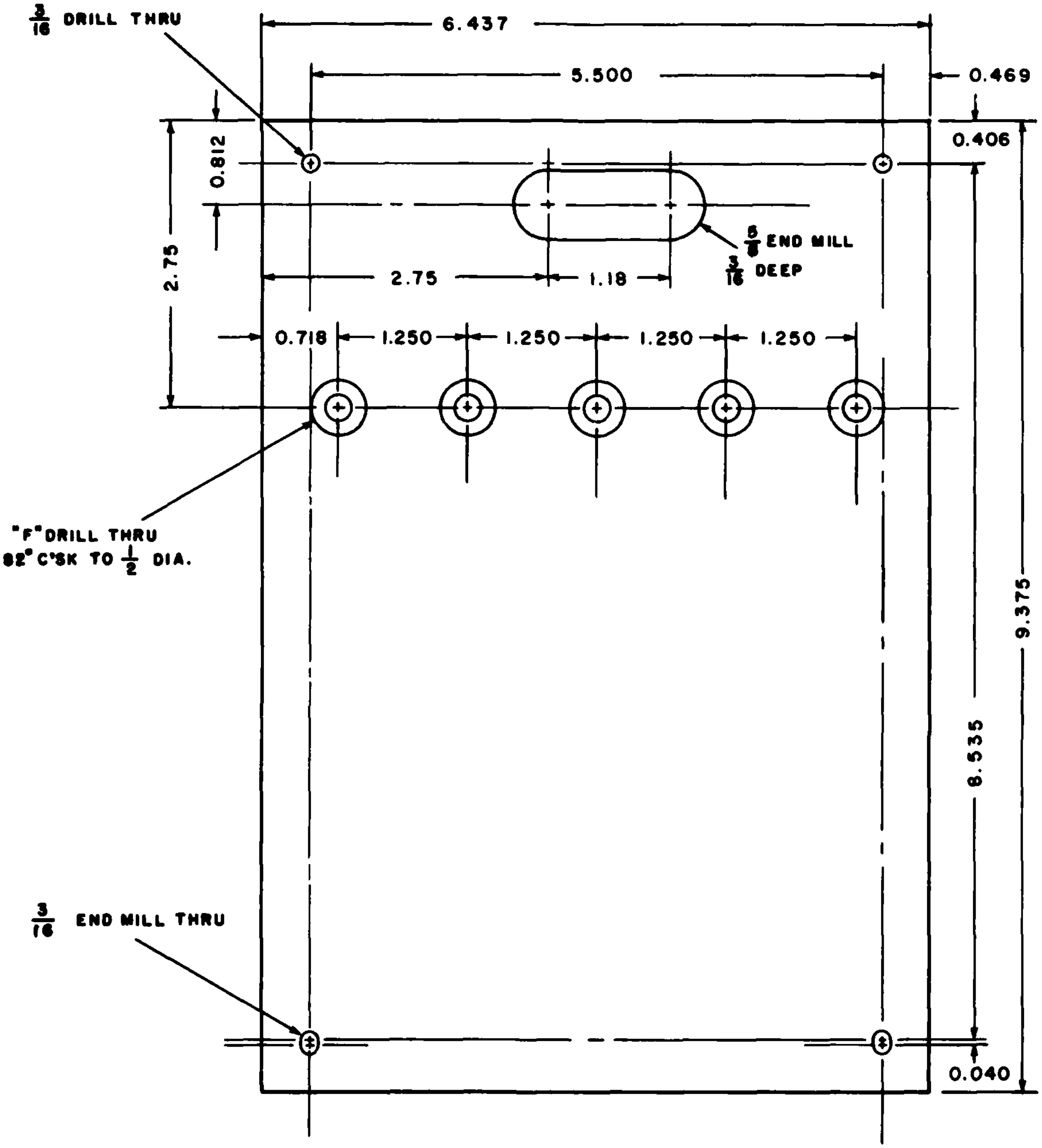
RESERVOIR CLAMP

MATERIAL - ALUMINUM

0 1 2

scale --- inches

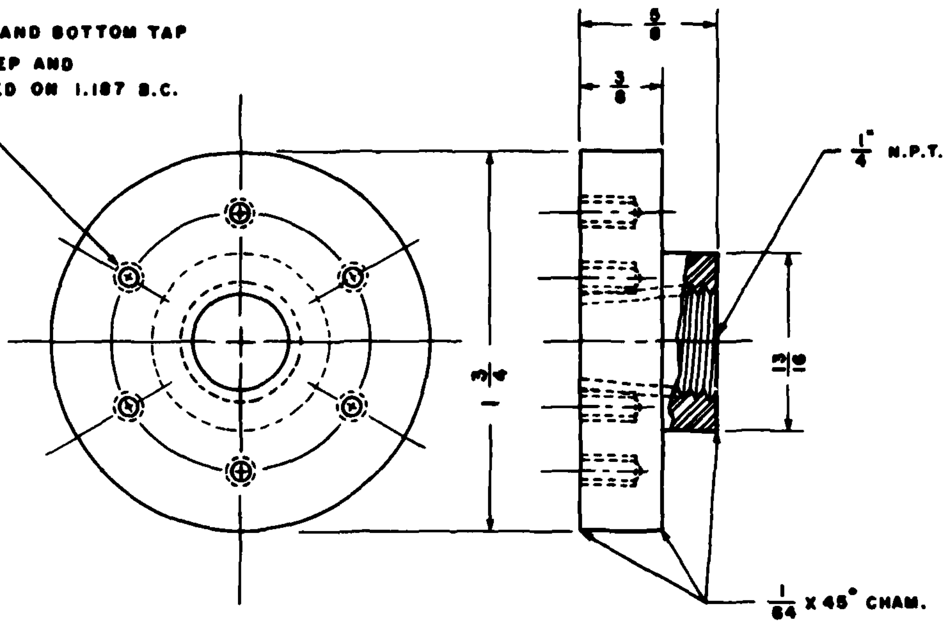
Appendix II-3



ADAPTOR PLATE

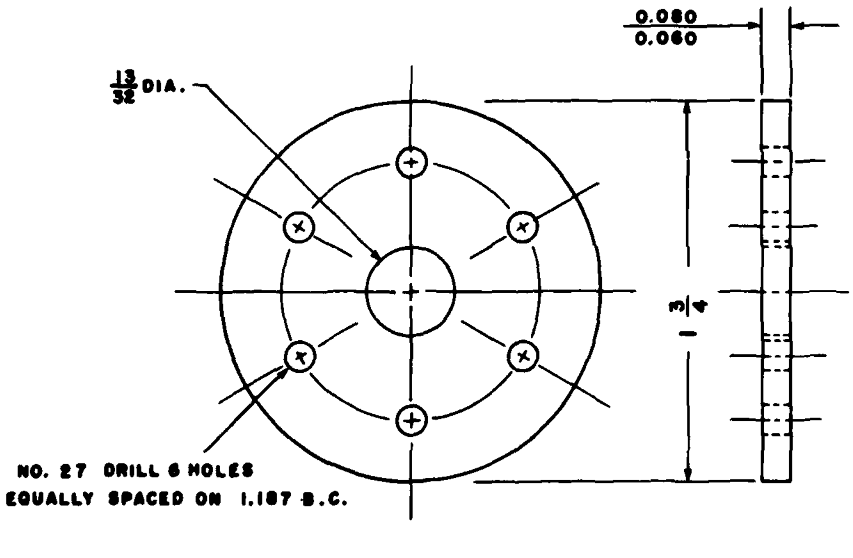
MATERIAL - 3/8" ALUMINUM

Appendix II-4

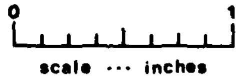


STAINLESS STEEL FLANGE

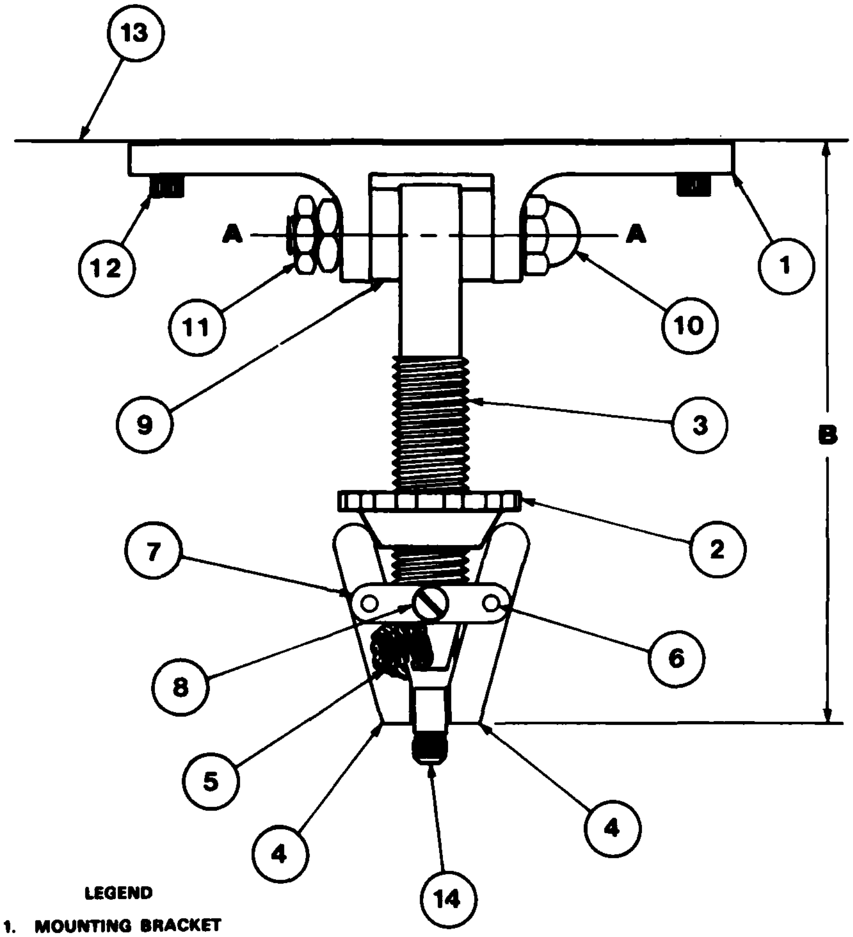
MATERIAL – 302 STAINLESS



TEFLON GASKET



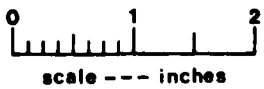
Appendix II-5



LEGEND

1. MOUNTING BRACKET
2. HANDWHEEL
3. THREADED SUPPORT
4. JAWS (2 Req'd)
5. JAW RETURN SPRING (2 Req'd)
6. JAW PIVOT (2 Req'd)
7. JAW MOUNT (2 Req'd)
8. JAW MOUNT FASTENER (2 Req'd)
9. SPACERS (2 Req'd)
10. ACORN NUT
11. HEX NUTS (2 Req'd)
12. SOCKET HEAD CAP SCREWS (2 Req'd)
13. TOP ENCLOSURE OF APPARATUS
14. NOZZLE OF APPARATUS

SAFETY CLAMP



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1 ORIGINATING ACTIVITY Defence Research Establishment Suffield	2a. DOCUMENT SECURITY CLASSIFICATION UNCLASSIFIED 2b. GROUP

3 DOCUMENT TITLE

Measurements of Elongational Viscometry Using a Fiber Spinning Technique
Part I: Modifications to the Sangamo Schlumberger Viscometer Model E4

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Suffield Report

5. AUTHOR(S) (Last name, first name, middle initial)

Gauthier Mayer, Michele D., Fenrick, Walter J., Armour, S. Joan

6. DOCUMENT DATE February 1984	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS 9

8a. PROJECT OR GRANT NO. 13E10	9a. ORIGINATOR'S DOCUMENT NUMBER(S) SR 377
8b. CONTRACT NO.	9b. OTHER DOCUMENT NO.(S) (Any other numbers that may be assigned this document)

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13. ABSTRACT (U)

The Sangamo-Schlumberger Elongational Viscometer (Model E4), as delivered by the manufacturer, did not maintain a constant elongational load under constant flow conditions and consequently could not be used to accurately measure elongational viscosity. The extensive modifications made to the instrument at DRES to correct this problem are described in detail as are other improvements made to the instrument.

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KEY WORDS

fiber spinning
elongational viscosity
Sangamo Schlumberger instrument
extensional viscosity
Newtonian liquids
rheology

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